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Low Temperature X-Ray Diffraction Techniques

I. REVIEW AND BIBLIOGRAPHY

II. A LOW TEMPERATURE SPECIMEN MOUNT
FOR THE SIEMENS HORIZONTAL DIFFRACTOMETER

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FOREWORD

This report was prepared by the Physics Laboratory. The work was initiated under Project No. 7360, "Materials Analysis and Evaluation Techniques," and Task No. 736005, "Compositional, Atomic, and Molecular Analysis." It was administered under the direction of the Directorate of Materials and Processes, Deputy for Technology, with W. L. Baun acting as project engineer.

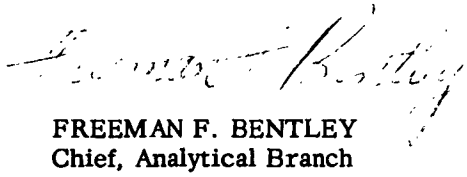
This report covers work conducted from March 1962 to February 1963.

The authors wish to acknowledge the help of Mark Goldschmidt on the bibliography. We compliment the excellent work of the Monsanto Research Corporation in the fabrication of the low temperature mount on a subcontract on contract no. AF 33(616)-7450.

ABSTRACT

A review and bibliography of 78 references on low temperature X-ray diffraction techniques are presented. A low temperature specimen mount for the horizontal Siemens diffractometer is described. Construction details, sampling techniques, and low temperature methods are discussed.

This report has been reviewed and is approved.



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I. REVIEW OF LOW TEMPERATURE TECHNIQUES

A study of materials at low temperatures requires specialized techniques and equipment. A review of literature in this area shows that investigators have proposed such techniques and equipment, some quite simple, while others are more sophisticated depending upon the specific nature of the problem and the desired results.

A number of investigators have proposed methods of adapting standard diffractometers and powder cameras for work in the low temperature region, while others have designed more specialized equipment and have described more elaborate experimental techniques.

Several publications reviewed presented adaptations of the Norelco Wide-Angle Diffractometer for low temperature investigations (refs 35, 40, 45, 56, 64, 75). These adaptations are generally quite simple in design and operation and inexpensive to fabricate. An example of such an attachment is shown in figure 1. The drawing illustrates a simple clip-on attachment for the Norelco diffractometer and incorporates a cooling system to be discussed. A good design feature shown in the drawing is the introduction of dry warm nitrogen into the space surrounding the sample chamber, eliminating the icing of the windows. These simple adaptors allow the study of materials in the temperature range from room temperature to liquid nitrogen temperatures (-190° C) by incorporating a cooling system of either liquid nitrogen or liquid-nitrogen-cooled gaseous nitrogen, or a combination of both. Such arrangements allow a variable controlled temperature range. In general, cooling is accomplished by passing dry gaseous nitrogen through a copper coil immersed in liquid nitrogen, after which the cooled gas is transported through an insulated or vacuum-jacketed delivery tube to the specimen chamber where it either passes through a sample block or holder or is "sprayed" directly onto the sample. By regulating the flow of the gas, the temperature may be controlled.

The actual cooling system may be as simple as just described, or it may be more complicated, and efficient, as the following examples. Figures 2 and 3 show arrangements which permit controlled temperature, continuous, unattended operation. In figure 2 the liquid nitrogen is boiled off by the insertion of a small resistance heater in the bottom of a 100 liter reservoir, thereby producing a continuous stream of cooled gaseous nitrogen. Figure 3 illustrates the more conventional design previously described with some modifications allowing continuous operation and temperature control. Liquid nitrogen in introduced through inlet tube U into a Dewar flask R in which is a copper coil C. Gaseous dry nitrogen is introduced through inlet tube N, and passes into a brass chamber H which serves as a cold trap for any impurities in the incoming gas. The nitrogen then passes into the copper coil and exits through tube O into the mixing head M. Here the cooled gas can be passed directly out through outlet B, or it can be mixed with warm dry gas introduced through O_1, thereby controlling the temperature. The nitrogen level in the Dewar is monitored by the float Z which is connected by a fine wire to a shutter of brass foil Q within the enclosed glass system E. The float and shutter rise and fall with the nitrogen level in the Dewar, the shutter operating between the two lamps L_1 and L_2 and the two photoelectric

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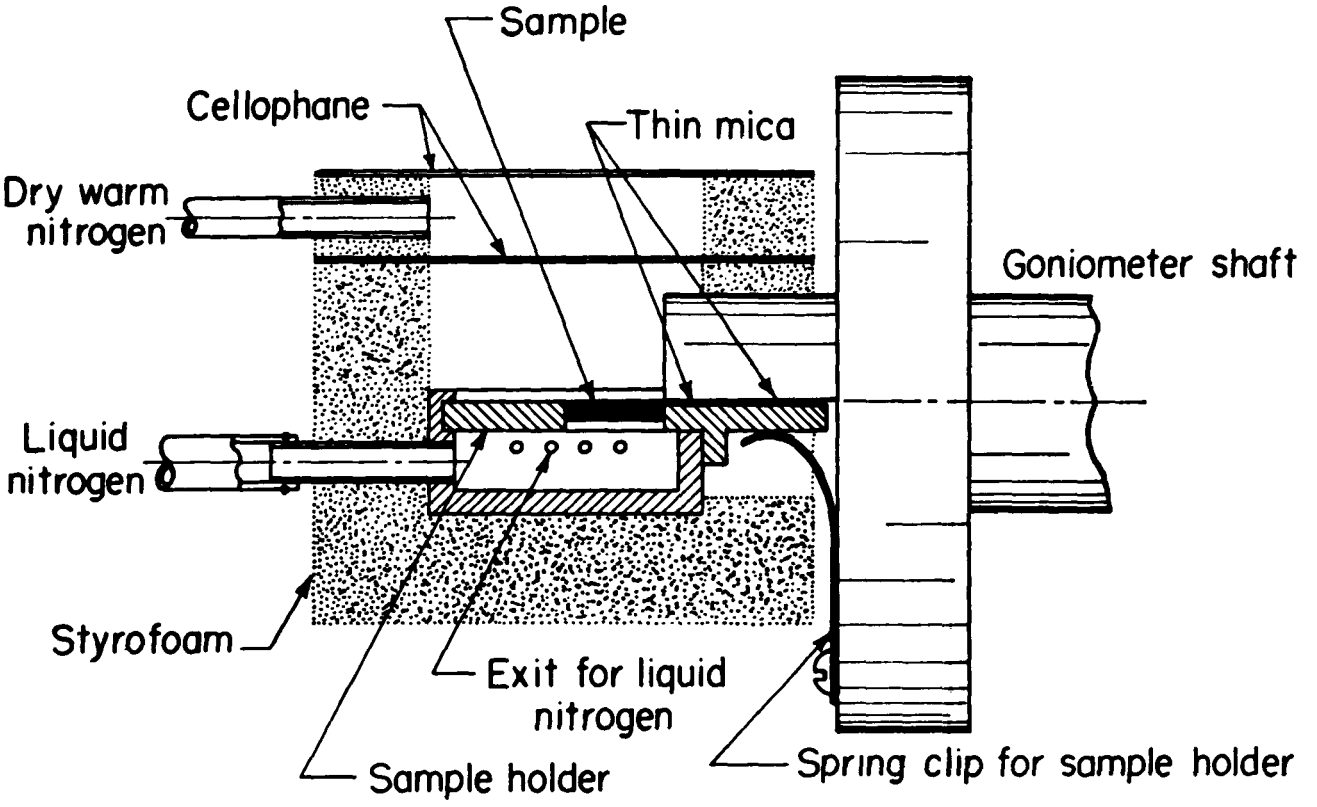


Figure 1. A Simple Low Temperature Diffractometer Mount

(From Calhoun, B.A. and Abrahams, S.C., "A Low Temperature Adaptor for the Norelco Wide Range Diffractometer," Review of Scientific Instruments, Vol 24, No. 5, May 1953)

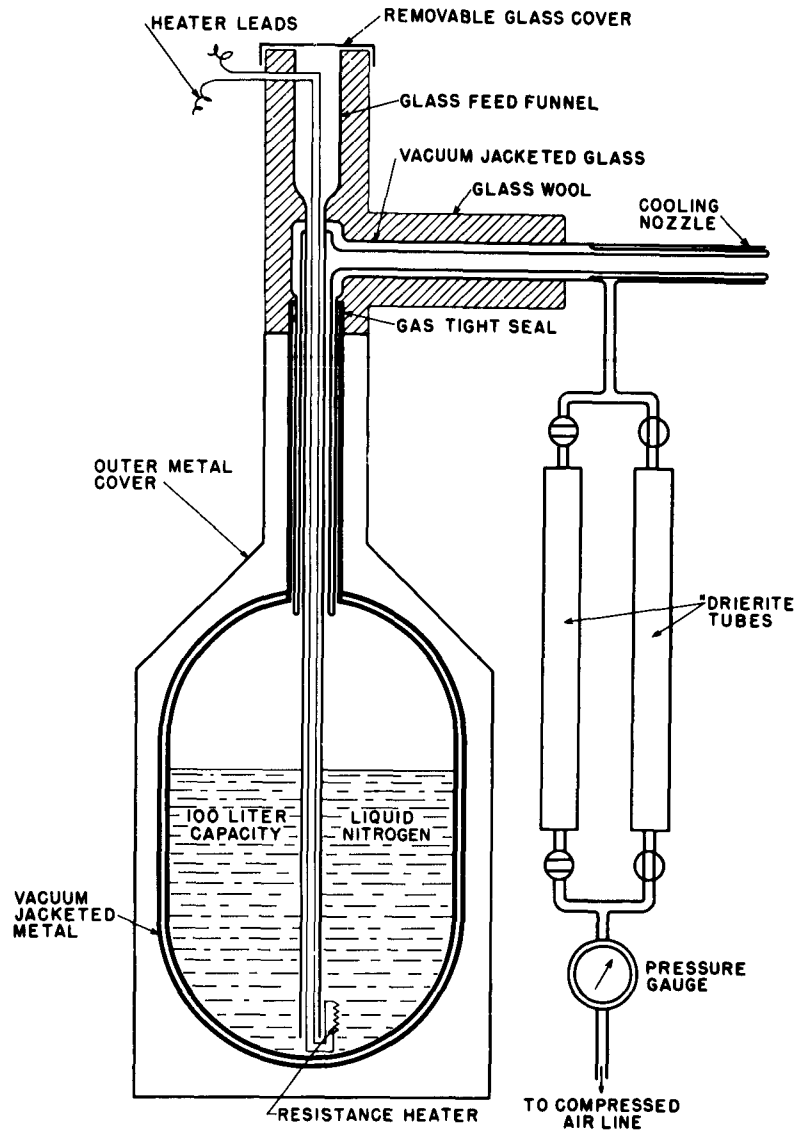


Figure 2. Cooling System for Continuous Operation

(From Burbank, R.D. and Bensey, F.N., The Application of the Buerger Precession Camera to Low Temperature Studies, Report No. K-841, Carbide and Carbon Chemicals Company, K-25 Plant, Oak Ridge, Tennessee, 1951)

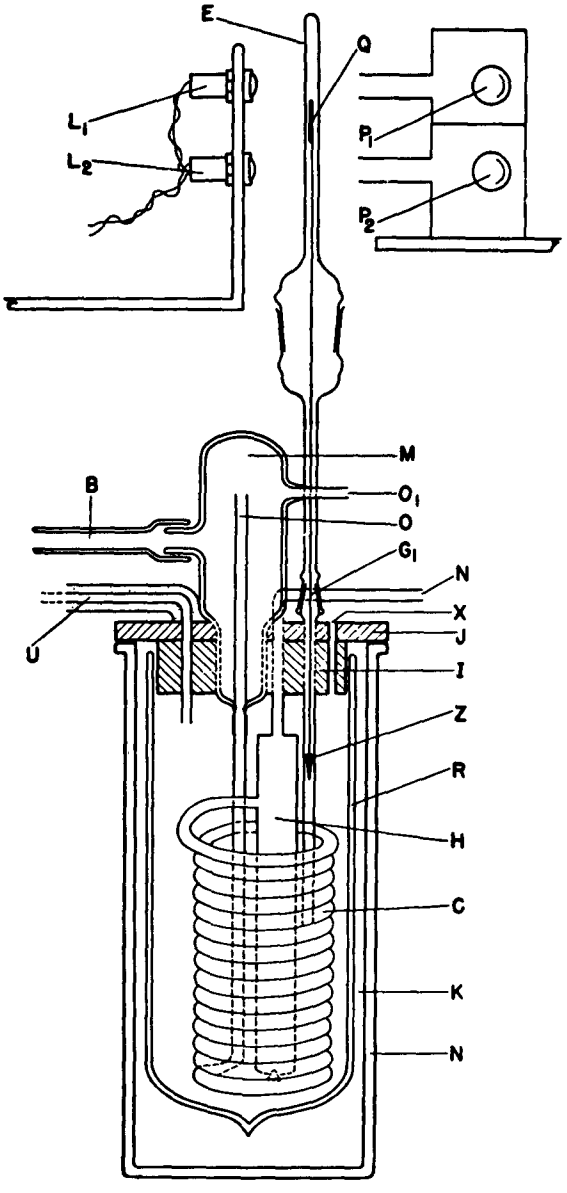


Figure 3. Cooling Chamber for Continuous Operation

(From Harding, T. T., "A Low-Temperature Apparatus for Single-Crystal X-Ray Goniometers," Canadian Journal of Chemistry, 34: 371-375, 1956)

cells P_1 and P_2. A circuit is used by which a drop in liquid level drops the shutter past the photocell P_2. This activates the cell which in turn activates a solenoid. The solenoid admits compressed air into a liquid nitrogen reservoir, forcing the liquid into the Dewar through filler tube U until the photocell P_1 is activated by the rising shutter, thereby shutting off the flow.

A much more sophisticated attachment for the diffractometer is shown in figure 4. The figure shows an exploded view of a low temperature vacuum attachment for the Norelco diffractometer. As can be seen in the drawing, the camera has its own self contained coolant flask, actually a modified Dewar, the sample being cooled by conduction. Various coolants can be used depending upon the range of temperature of interest. The temperature is controlled by heating the sample block while the flask is kept full of coolant.

The gaseous refrigerant method of cooling is readily adaptable to the standard powder camera, allowing photographic recording of low temperature diffraction effects (refs 6, 12, 13, 18, 19, 23, 32, 33, 34, 36, 39, 41, 49, 52, 55, 61, 66, 69, 74). A simple powder camera arrangement is illustrated in figures 5 and 6, incorporating a stream of cooled gas as a refrigerant. In figure 6, B represents alignment pins which aid in the assembly of the camera while C is the thermocouple. Several other camera designs, cooling systems, and specimen mountings are described in the references. The ability to evacuate the camera is a distinct advantage in that it eliminates the icing problem and should be incorporated when possible. In designing a low temperature powder camera, an effort should be made to isolate the film from the cooled area (ref 13) or at least to insulate the film from the direct cooling action of the refrigerant by incorporating a suitable shield, since low temperature severely affects the sensitivity of X-ray film.

A more sophisticated arrangement is the design of a metal cold cell or cryostat employing either liquid nitrogen or helium or both as a coolant. Cryostats are normally mounted on a diffractometer and provide either photographic or electronic detection. Such a design is presented by the authors and is described in detail in section II. Several other cryostat designs have been reviewed and are all of the same general design (refs 11, 54, 58, 73, 76). In this arrangement, a metal Dewar is bolted to a chamber fitted with a window. A sample block is attached to the lower end of the inner bucket of the Dewar and extends down into the lower chamber where it intersects the X-ray beam. The interior of the instrument is evacuated and the sample cooled by conduction. The device allows the study of not only solid and powder samples, but also liquids and gases which may be admitted into the specimen chamber by a suitable method, where they condense on the cold sample block. The design of a helium cryostat is more complicated. To increase the efficiency and to preserve the coolant the helium bucket must be surrounded by a separate nitrogen bucket, the entire assembly contained in a high vacuum (ref 76). Authors have proposed many special design and fabrication features which certainly should be considered if the expense permits.

Low temperature techniques are not restricted to diffractometer and powder camera applications, but are readily adaptable to single crystal studies. For such investigations of single crystals at low temperatures, both the Weissenberg and Buerger precession cameras can be adapted for studies down to liquid nitrogen temperatures (refs 26, 30, 37, 38, 46, 51, 53, 59, 68, 70, 71). Low temperature investigations are particularly advantageous in the case of single crystals because of the increase of intensity of the reflections.

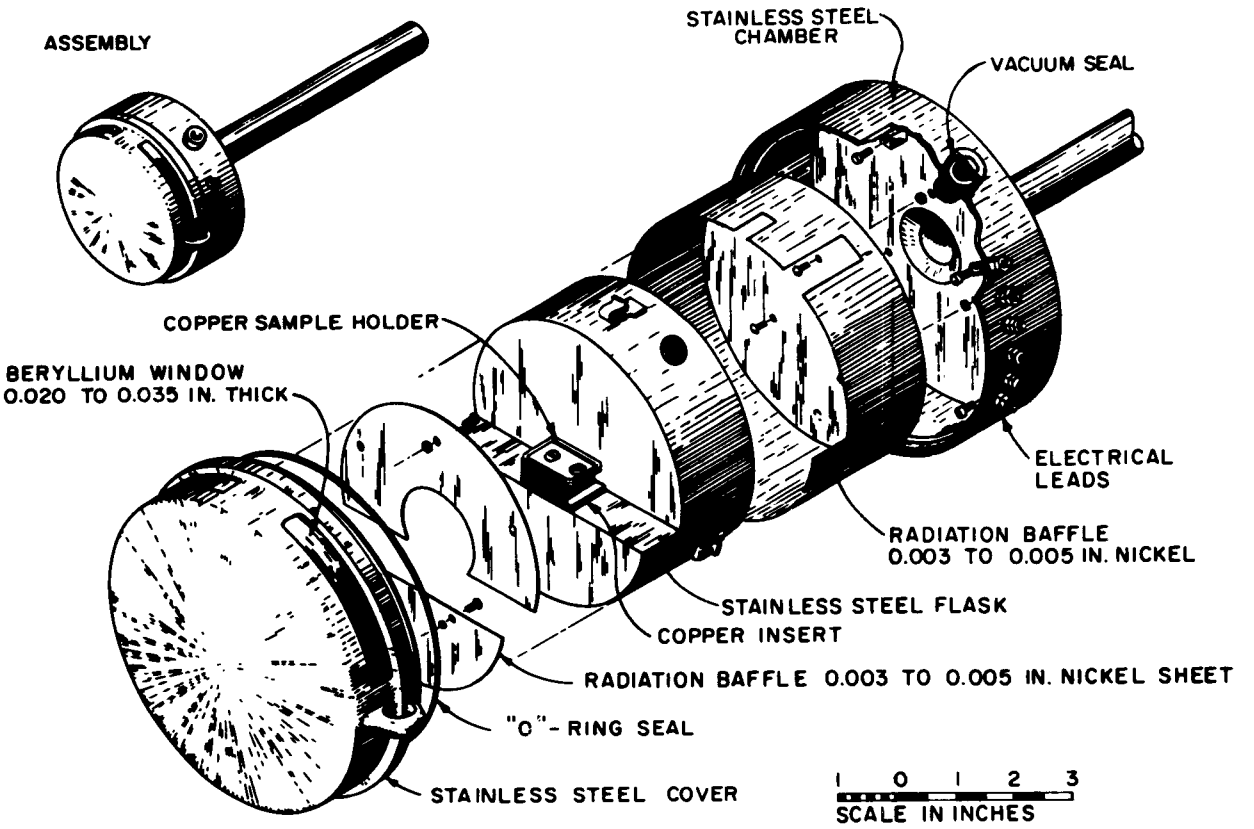
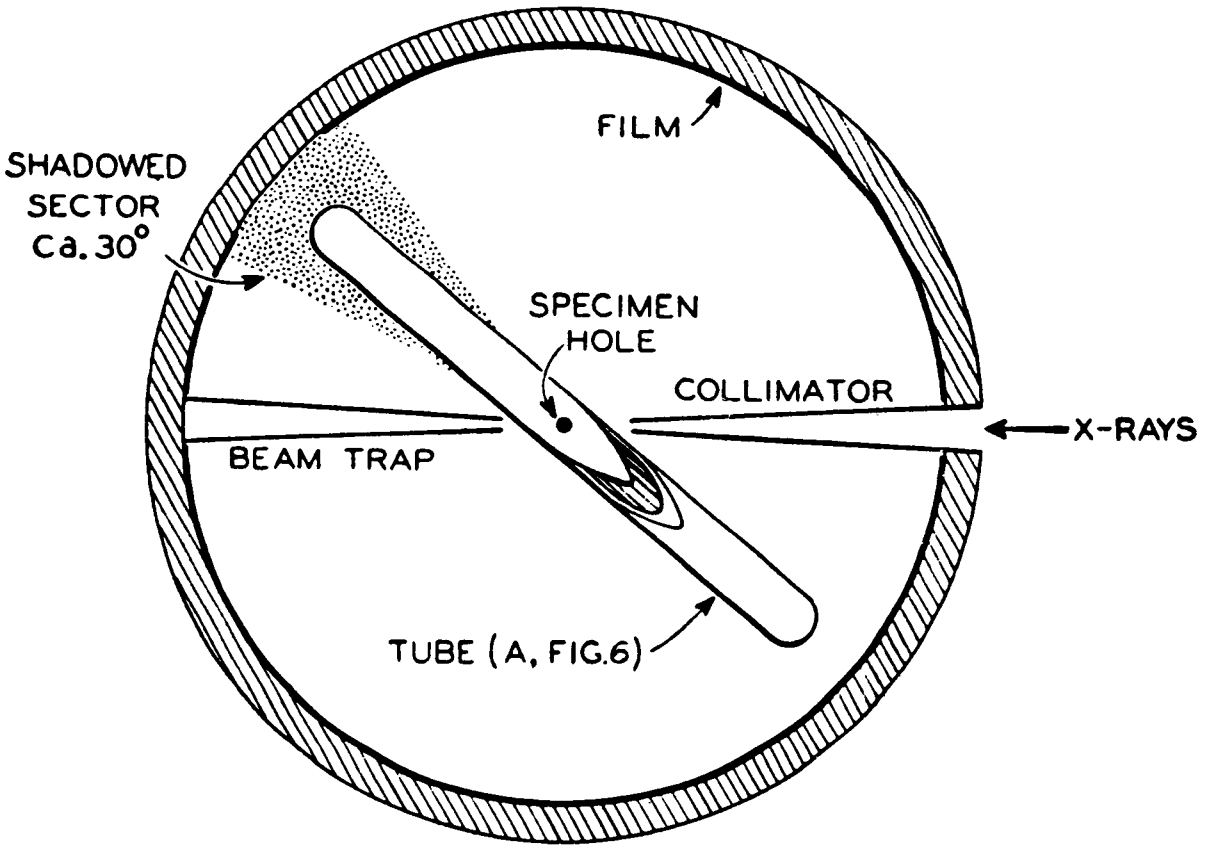


Figure 4. Exploded Drawing of Low Temperature Camera

(From Jetter, L. K., McHargue, C. J., Williams, R. D., and Yakel Jr., H. L., "Low-Temperature Camera for X-Ray Diffractometer," Review of Scientific Instruments, Vol 28, No. 12, 1087-1088, Dec 1957)



Diagrammatic sketch showing relation of cooling tube to x-ray beam and film. View from base of specimen along rotation axis.

Figure 5. Diagrammatic Sketch of Low Temperature Powder Camera

(From Wood, E.A., "Sample Attachment for Low Temperature Use of an X-Ray Diffraction Camera," Review of Scientific Instruments, Vol 24, No. 4, 325-326, April 1953)

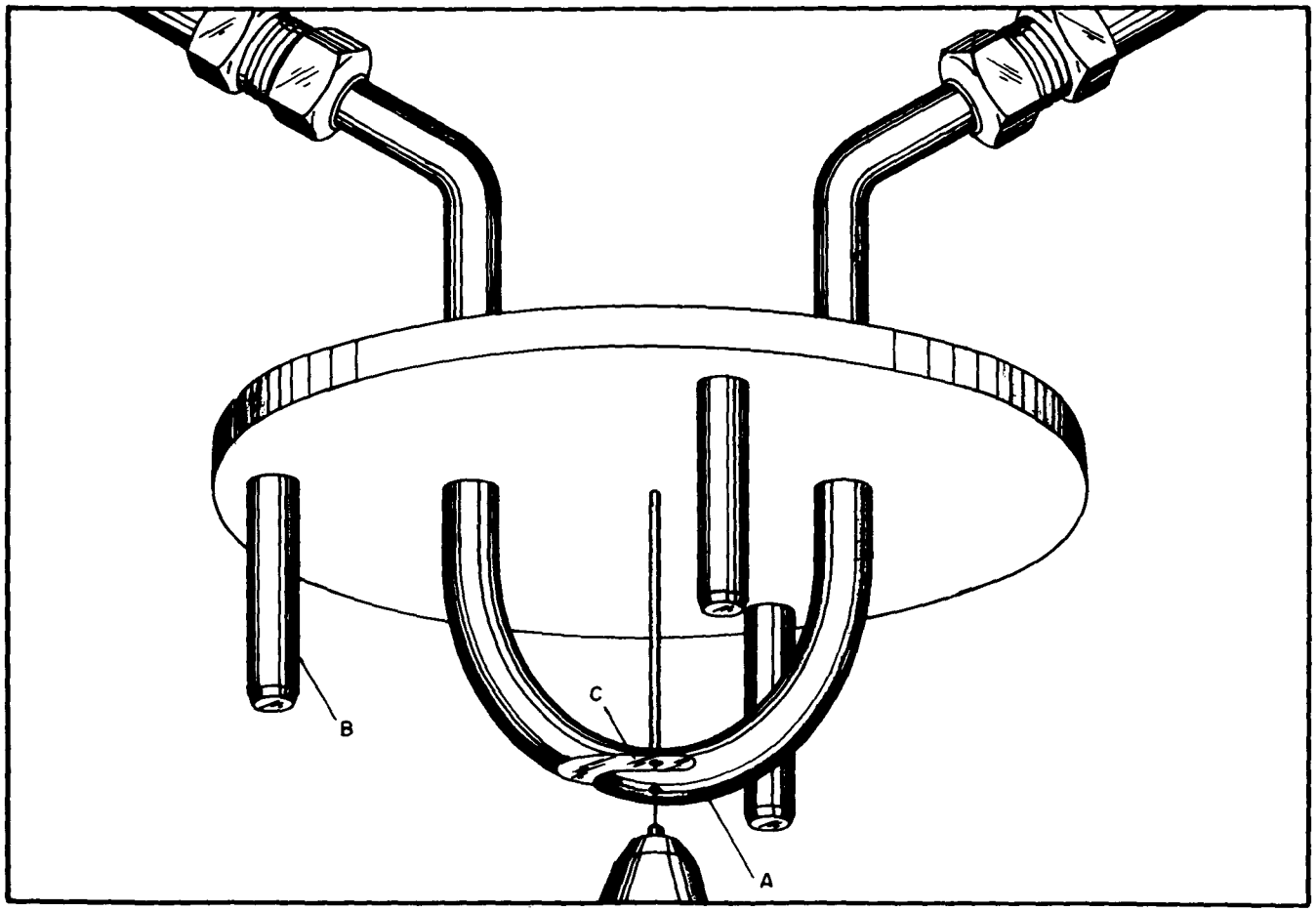


Figure 6. Cooling System of Low Temperature Powder Camera

(From Wood, E.A., "Simple Attachment for Low Temperature Use of an X-Ray Diffraction Camera," Review of Scientific Instruments, Vol 24, No. 4, 325-326, April 1953)

The coolant delivery system in the single crystal arrangements generally follows that illustrated in figure 7. The figure shows the two main features of such a system. The central double-walled, evacuated coolant delivery tube is surrounded at the delivery end by an open jacket through which is passed warm, dry gas. The warm gas serves as an effective shield and insulator for the central cooled stream of gas as it bathes the sample. A more detailed drawing is shown in figure 8. This general scheme can be readily applied to any single crystal camera.

Low temperature techniques are used for a great number of practical and experimental applications. One very important and common application of low temperature diffraction is the determination of thermal expansion and symmetry changes of materials at low temperatures (refs 3, 10, 20, 21, 22, 27, 57, 65). Thermal properties and phase transformations can be accurately and easily determined by standard methods in the range from room temperature to the temperatures of liquid nitrogen or helium. Techniques have also been proposed to study materials at low temperatures and variable pressures (ref 17).

An important metallurgical application is the structural examination of metals upon deformation at low temperatures (refs 47, 50, 67, 72). Such methods allow the study of structural changes due to cold working, stretching, etc., in situ at these low temperatures.

Several techniques have been proposed for the X-ray study of solidified gases (refs 4, 5, 7, 14, 15) and liquids (ref 48).

Thermocouples suitable for use at these low temperatures have been investigated and reported (ref 42).

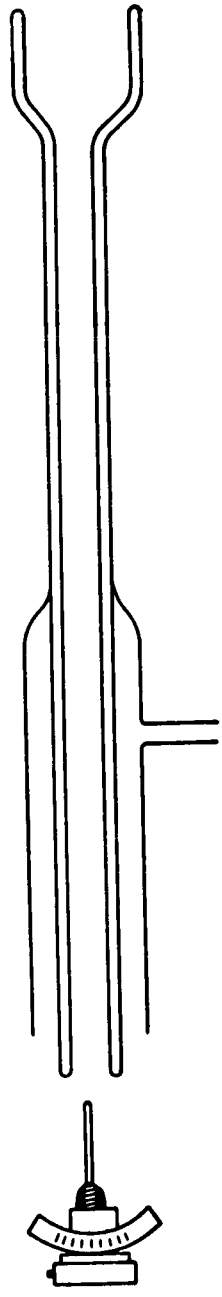


Figure 7. Delivery Tube for Single Crystal Cooling System
(From Harding, T. T., "A Low-Temperature Apparatus for Single-Crystal X-Ray Goniometers," Canadian Journal of Chemistry, 34: 371-375, 1956)

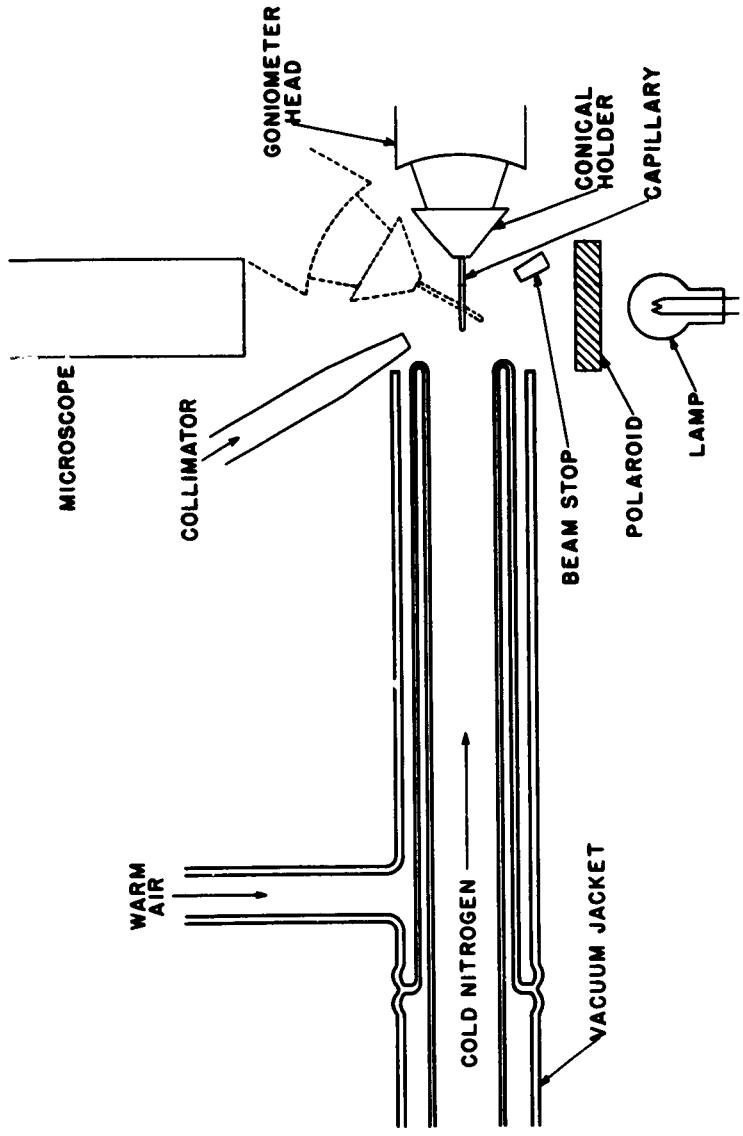


Figure 8. Low Temperature Arrangement for Precession Camera
(From Burbank, R.D. and Bensey, F.N., The Application of the Buerger Precession Camera to Low Temperature Studies, Report No. K-841, Carbide and Carbon Chemicals Company, K-25 Plant, Oak Ridge, Tennessee, 1951)

II. A LOW TEMPERATURE SPECIMEN MOUNT FOR THE SIEMENS HORIZONTAL DIFFRACTOMETER

A low temperature specimen mount for the horizontal Siemens X-ray diffractometer is described. It was necessary to design such a specimen mount because the literature does not describe a mount which fits the needs of the Physics Laboratory. As was shown in the review, literature designs are primarily single crystal apparatus, film cameras, or designs for the vertical diffractometer of the Philips type. One excellent design for a horizontal diffractometer mount (ref 58) is far too complicated for our purposes and probably too large to fit the Siemens diffractometer.

Description of the Mount

Figure 9 shows an exploded view of the specimen mount for low temperatures. At the bottom of the figure is seen the base which bolts tightly to the diffractometer through the same three holes that normally hold the specimen holder for room temperature work. Fitting this base is the vacuum enclosure and window assembly, on which are mounted the pumping and venting ports, alignment screws, and alignment bars. The window is 10 mil beryllium curved to the radius of the enclosure and is cemented in with epoxy resin cement. The windows cover slightly more than 180° to allow for alignment at zero degrees 2θ. Fitting on to this vacuum tight enclosure is the stainless steel Dewar. The bottom of the inner bucket of this Dewar is copper with a copper button extending up into the liquid nitrogen or other coolant. The copper specimen holder bolts tightly to the copper block on the bucket and is pinned so that it must be properly aligned in order to fit on the block. The Dewar is also pinned so that it may be placed on the enclosure and always be in alignment. An "O" ring furnishes the seal between the Dewar bucket and the enclosure. An insulated two-conductor vacuum seal is used for bringing the iron - constantan thermocouple into the chamber.

Figure 10 shows the low temperature mount on the Siemens diffractometer. Note that the alignment bar fits into the jig used for aligning the original room temperature mount. Figure 11 shows a similar view of the mount with the Dewar removed to show the interior of the assembled mount.

Three different configurations have been used for sample holders. One is machined from copper and the sample is placed directly on the copper specimen surface. Another accommodates a frosted glass microscope slide and still another has a $\frac{5}{8}$ inch hole and accommodates bulk rods or buttons.

Alignment

The mount is aligned by first aligning the diffractometer on zero degrees 2θ using standard Siemens techniques. Then, the sample block on the bottom of the Dewar bucket is replaced by a zero alignment device as shown in figure 12. This device is identical to the sample holder except that it holds the Siemens zero alignment slide which is two pieces of glass separated by a thin foil. When properly aligned, a very thin unattenuated X-ray beam will reach the counter at zero degrees 2θ. When this beam is maximized, the diffractometer and sample holder are aligned at zero degrees 2θ. Now the zero alignment device is removed and the copper sample holder is attached to the Dewar bucket. The position of the first line of copper is checked and, if it is not exactly correct, the diffractometer or tube take-off angle may be changed to bring the position of the line to the proper place. If the glass slide holder is to be used, the gold standard slide furnished by Siemens may be used for alignment rather than the copper holder.

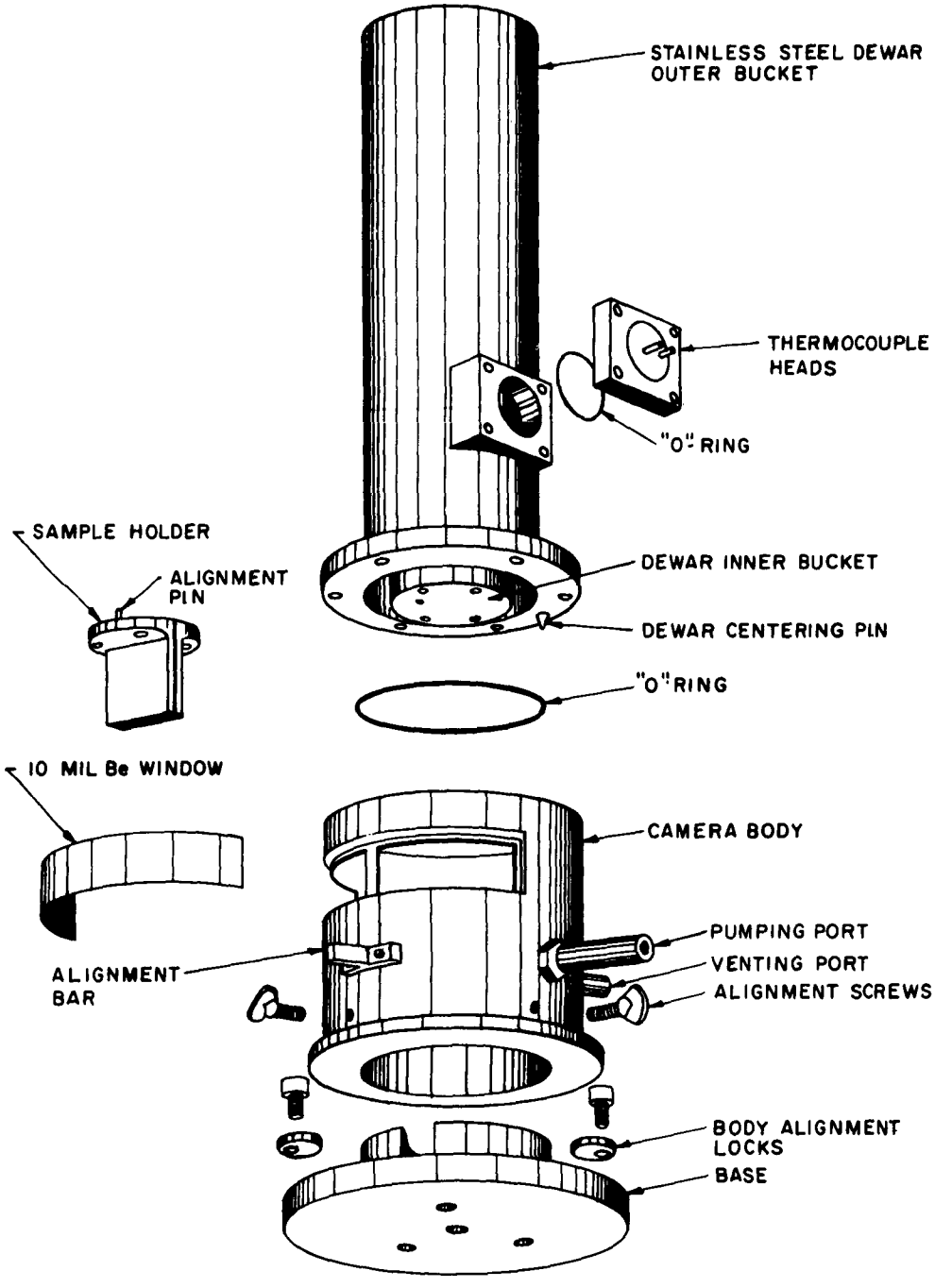


Figure 9. Exploded View of the Low Temperature Mount

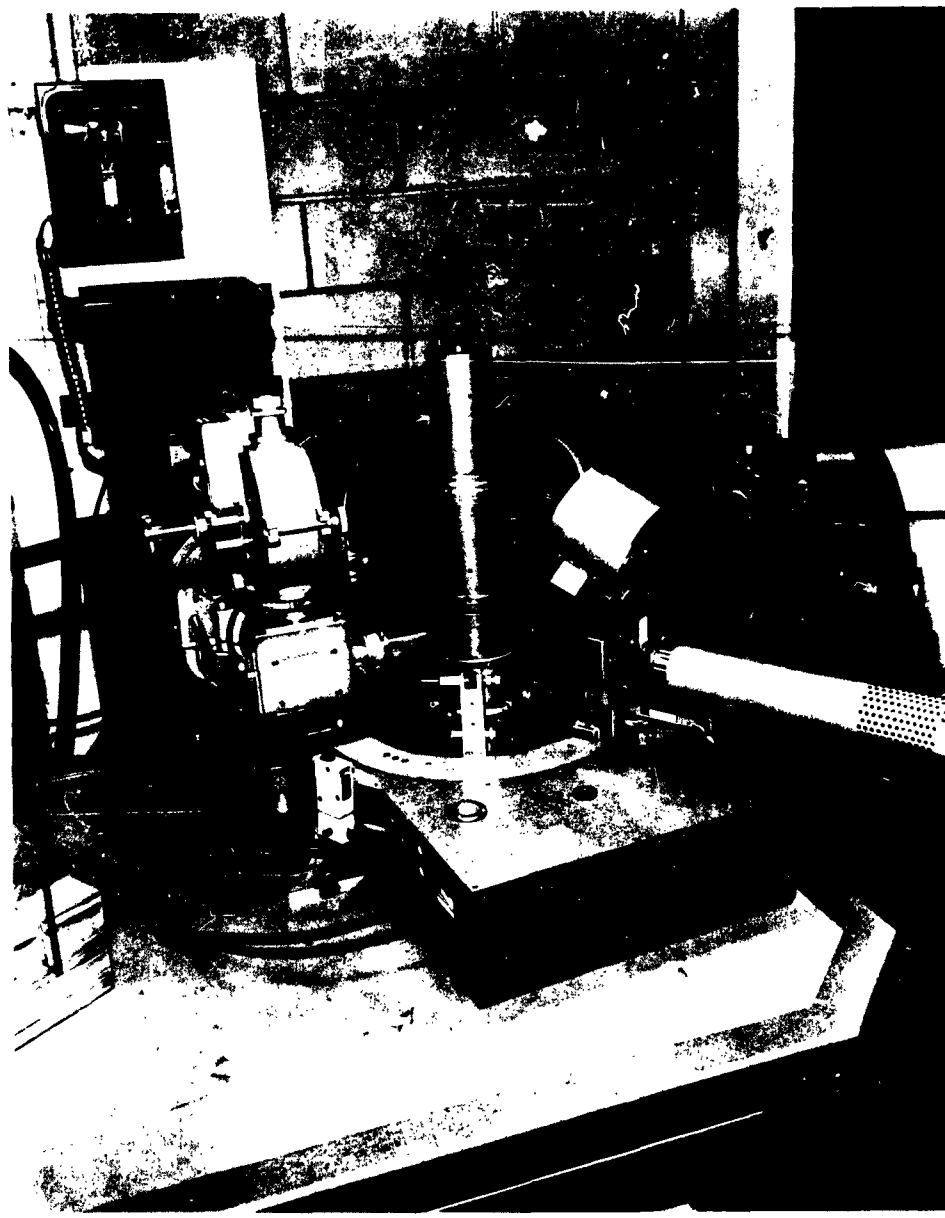


Figure 10. The Low Temperature Mount Assembled on the Diffractometer

Figure 11. The Low Temperature Mount With the Dewar Removed From the Base

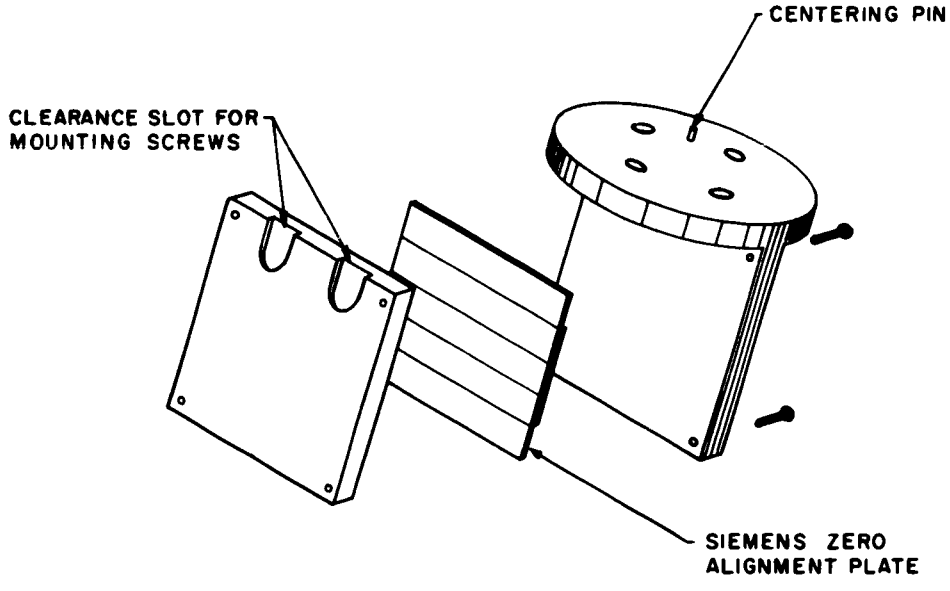


Figure 12. Zero Alignment Device

Obtaining Low Temperatures

Normally for routine work in identifying organic liquids, the sample is placed on the sample holder and the Dewar quickly chilled to crystallize the sample. The Dewar is then placed on the enclosure and a rough vacuum of about 25μ is pulled on the system to prevent icing and to act as an insulator for the Dewar vessel. With liquid nitrogen at -196° C in the Dewar, the specimen temperature at the center of the specimen is -190° C. Thermal gradients across the sample, therefore, should be no more than 5° C. For higher temperatures, a dry ice (CO_2) - acetone mixture works well. If a number of different temperatures are necessary, then a cooling coil could be placed in the Dewar with an appropriate heat exchanging liquid and a flow of liquid or cooled gaseous nitrogen could be maintained to obtain any temperature from room temperature to -190° C. For temperatures above room temperature, the same technique may be used by heating the circulating gas and using silicone oil in the Dewar as the heat exchanging medium. This mount has been used up to 100° C with an immersion heater and silicone oil. Temperatures much higher than 100° C could not be obtained with this mount because soft solder was used to solder the copper block to the bottom of the Dewar bucket. If temperatures higher than 100° C were to be achieved, silver solder could be used.

To illustrate the typical use of such a low temperature specimen mount, figure 13 was prepared showing the powder pattern for acetonitrile at -190° C and at -75° C. As can be seen, a phase change has occurred between -190° C and -75° C (actually several phases exist in acetonitrile and will be discussed in a later report). This result correlates directly with effects shown in EPR spectroscopy by L. A. Harrah of the Physics Laboratory.

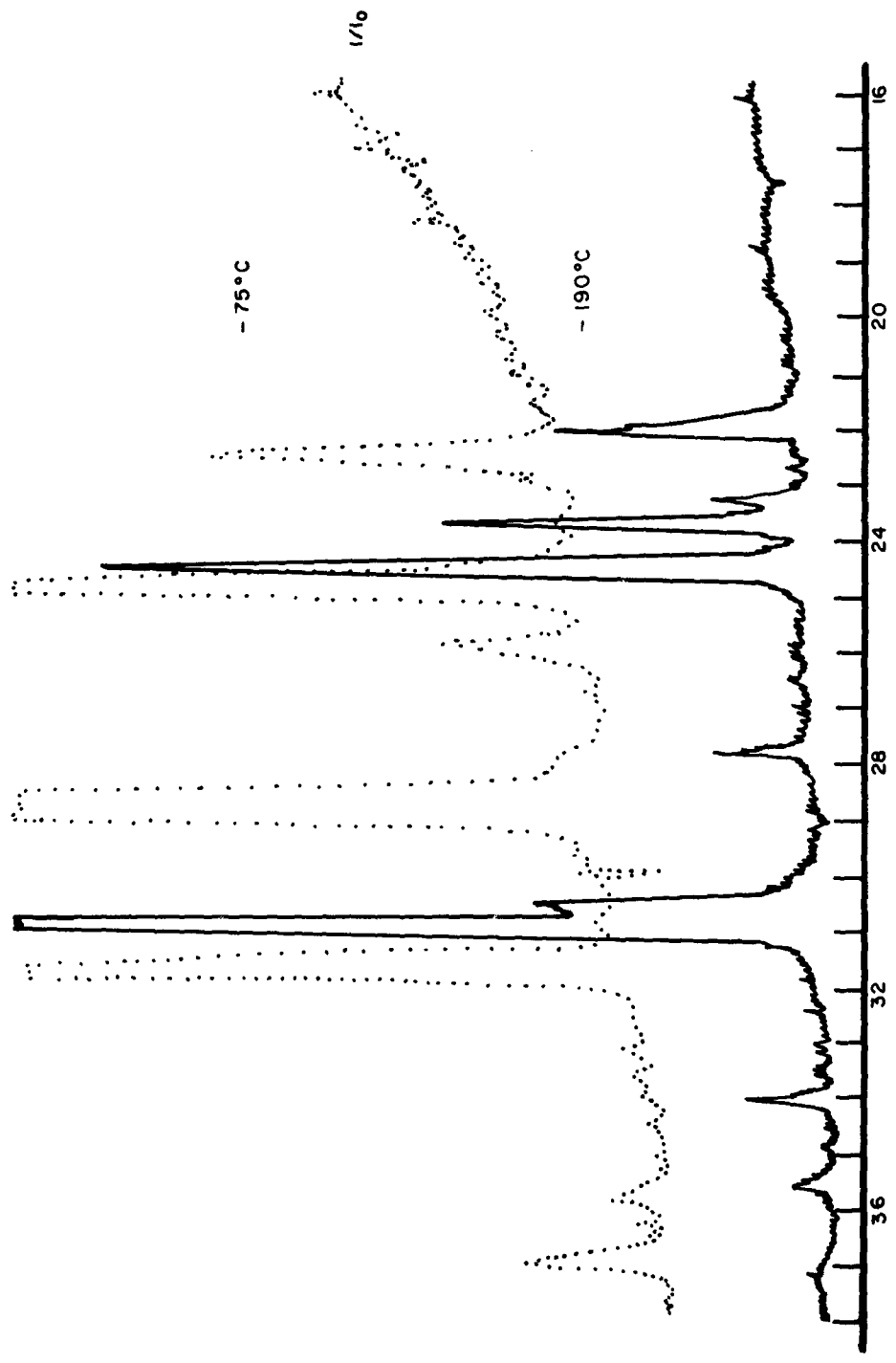


Figure 13. Powder Pattern for Acetonitrile at -190°C and -75°C

REFERENCES AND BIBLIOGRAPHY ON

LOW TEMPERATURE TECHNIQUES AND APPARATUS

Prior to 1950

1. Cioffi, P. P. and Taylor, N. W., "A Method of Maintaining Small Objects at Any Temperature Between -180° and +20°," J. Optical Soc. Am. 6, 906-9 (1922).

2. Keesom, W. H., "The Diffraction of X-Rays by Fused Sodium and Potassium," Verslag Akad. Wetenschappen Amsterdam 35, 1141-3 (1926).

3. McLennan, J. C. and Monkman, R. J., "Thermal Expansion of Zinc and Cadmium Crystals and the Crystal Structure of Erbium and Columbium," Trans. Roy. Soc. Can., Sect. III, 23, 255-67 (1930).

4. Natta, I. G. and Nasini, A. G., "The Crystal Structure of Xenon," Nature 125, 457 (1930).

5. Mooy, H. H., "Crystal Structure of Methane, II," Proc. Acad. Sci. Amsterdam 34, 660-2 (1931).

6. Taylor, N. W., "An X-Ray Camera for Powder Diagrams at Any Temperature," Rev. Sci. Instr. 2, 751-5 (1931).

7. Vegard, L., "Structure of Solid N_2O_4 at the Temperature of Liquid Air," Z. Physik 68, 184-203 (1931).

8. Santos, J. A. and West, C. J., "A Method of Taking X-Ray Photographs of Crystalline Powders at the Temperature of Liquid Air," J. Sci. Instr. 10, 219-21 (1933).

9. Grime, H. and Santos, J. A., "The Structure and Color of Anhydrous Cobalt Chloride at Room and Very Low Temperatures," Z. Krist. 88, 136-41 (1934).

10. Keesom, W. H. and Kohler, J. W. L., "The Lattice Constant and Expansion Coefficient of Solid Carbon Dioxide," Physica 1, 655-8 (1934).

11. Pohland, E., "A Handy X-Ray Vacuum Camera for Any Low Temperature," Z. Physik Chem. B26, 238-45 (1934).

12. Keesom, W. H. and Taconis, K. W., "An X-Ray Goniometer for Investigation of the Crystal Structure of Solidified Gases," Physica II, 463-71 (1935).

13. Ruheman, B., "New Camera for Low-Temperature X-Ray Diffraction Work," Physik Z. Sowjetunion 7, 572-82 (1935).

14. Vonnegut, B. and Warren, B. E., "The Structure of Crystalline Bromine," J. Am Chem. Soc. 58, 2459-61 (1936).

15. Keesom, W. H. and Taconis, K. W., "Structure of Solid Helium," Physica 5, 161-9 (1938).

16. Reekie, J., "Scattering of X-Radiation by Liquid Helium II," Proc. Cambridge Phil. Soc. 36, 236-41 (1940).

17. Wittstadt, W., "X-Ray Photography under Extreme Conditions," Z. Elektrochem 46, 521-7 (1940).

18. Lonsdale, K. and Smith, H., "X-Ray Crystal Photography at Low Temperatures," J. Sci. Instr. 18, 133-5 (1941).

19. Venkateswaran, C. S., "Low-Temperature Studies of the Raman X-Ray Reflections in Crystals," Proc. Indian Acad. Sci. 14A, 387-94 (1941).

20. Ubbelohde, A. R. and Woodward, I., "Structure and Thermal Properties of Crystals. V. Thermal Expansion of Phthalocyanines and Porphines," Proc. Roy. Soc. A181, 415-27 (1943).

21. Ubbelohde, A. R. and Woodward, I., "Structure and Thermal Properties of Crystals. VI. The Role of the Hydrogen Bonds in Rochelle Salt," Proc. Roy. Soc. A185, 448-65 (1946).

22. Ubbelohde, A. R. and Woodward, I., "Structure and Thermal Properties Associated with Some Hydrogen Bonds in Crystals. VII. Behavior of KH_2PO_4 and KH_4AsO_4 on Cooling," Proc. Roy. Soc. 188, 358-71 (1947).

23. Vegard, L., "Investigation into the Structure and Properties of Solid Matter with the Help of X-Rays," Strifter Norske Videnskaps Akad. Oslo I. Mat-Naturv, Klasse, No. 2 (1947).

24. Kaufman, H. and Fankuchen, I., "Some Cameras for the Study of Crystals at Low Temperatures," ASXRED Winter Meeting Battelle Memorial Institute, Columbus, Ohio (1948).

25. Bouttier, L., "Apparatus for Obtaining and Transporting Single Crystals at Low Temperatures. Applications to the Acid HNO_3 to Its Hydrates and Their X-Ray Diagrams," C. R. Acad. Sci. 228, No. 18, 1419-21, (1949).

26. Kaufman, H. S. and Fankuchen, I., "A Low-Temperature Single-Crystal X-Ray Diffraction Technique," Rev. Sci. Instr. 20, 733-4 (1949).

27. Kay, H. F. and Vousden, P., "Symmetry Changes in Barium Titanate at Low Temperatures and Their Relation to Its Ferroelectric Properties," Phil. Mag. 40, 1019-40 (1949).

1950

28. Clifton, D. F., "Low Temperature X-Ray Diffraction Apparatus," Rev. Sci. Instr. 21, 339-42.

29. Frazer, B. C. and Pepinsky, R., "Low Temperature X-Ray Diffraction Goniometer,' Phys. Rev. 80, 124.

1951

30. Burbank, R. D. and Bensey, F. N., "The Application of the Buerger Precession Camera to Low Temperature Studies," Carbide and Carbon Co., Oak Ridge, Tenn.

31. Post, B., Schwartz, R. S. and Fankuchen, I., "Improved Device for X-Ray Diffraction Studies at Low Temperatures," Rev. Sci. Instr. 22, 218-19.

1952

32. Dunoyer, J. M., "Debye-Scherrer Diagrams of Compounds Crystallizing at Low Temperatures. Application to Alcohols," Helv. Chim. Acta 35, 840-5.

33. Tombs, N. C., "A Low-Temperature X-Ray Powder Camera," J. Sci. Instr. 29, 364.

1953

34. Bouttier, L. and Dunoyer, J. P., "Description of a Photographic Chamber for Taking Debye-Scherrer Diagrams of Specimens at Very Low Temperatures," Bull. Soc. Mineral 76, 1-3, 79-85.

35. Calhoun, B. A. and Abrahams, S. C., "A Low Temperature Adaptor for the Norelco Wide Range Diffractometer," Rev. Sci. Instr. 24, No. 5.

36. Hutchinson, C. A., Jr. and Miller, C. C., "A Low Temperature X-Ray Diffraction Camera," Bull. Amer. Phys. Soc. 3, No. 4, 304.

37. Keeling, R. O., Jr., Frazer, B. C. and Pepinsky, R., "Low Temperature X-Ray Goniometer for Structural Studies of Crystal Transitions," Rev. Sci. Instr. 24, 1087-95.

38. Steinfink, H., Ladell, J., Post, B. and Fankuchen, I., "A Low Temperature Weissenberg X-Ray Camera," Rev. Sci. Instr. 24, 882-3.

39. Wood, E. A., "Simple Attachment for Low Temperature Use of an X-Ray Diffraction Camera," Rev. Sci. Instr. 24, 325-6.

1954

40. Calhoun, B. A. and Abrahams, S. C., "A Low Temperature Adaptor for the Norelco Wide Range Diffractometer," Norelco Reporter I, No. 3, Reprinted from Rev. Sci. Instr. 24 (1953).

41. Owen, E. A. and Williams, G. I., "Low Temperature X-Ray Camera," J. Sci. Instr. 31, 49-54.

42. Pearson, W. B., "Thermocouples Suitable for Use at Low Temperatures," J. Sci. Instr. 31, 444.

43. Tweet, A. G., "Small-Angle X-Ray Scattering from Liquid Helium I and Liquid Helium II," Phys. Rev 93, 15-20.

44. Wallwork, S. C. and Harding, T. T., "A Simple Method of Obtaining Low-Temperature X-Ray Diffraction Photographs," J. Sci. Instr. 31, 163-4.

1955

45. Butters, R. G. and Myers, H. P., "A Low Temperature Attachment for an X-Ray Goniometer," Canad. J. Technology 33, 356-9.

46. Kreuger, A., "A Weissenberg Camera for Use at Constant Temperatures Between about -150° and 300°," Acta Cryst. 8, 348.

47. Pearson, W. B., "A Method of Examining Structural Changes of Metals on Deformation in Liquid Helium: Examination of Indium," Can. J. Phys. 33, 473-82.

48. Smith, H. N. and Heady, H. H., "Identification of Frozen Liquid Samples with the X-Ray Diffractometer," Anal. Chem. 27, 883-8.

49. Thewlis, J. and Davey, A. R., "Adaption of a Standard X-Ray Powder Camera for Work at Low Temperatures," J. Sci. Instr. 32, No. 2, 79.

1956

50. Barrett, C. S., "X-Ray Study of the Alkali Metals at Low Temperatures," Acta Cryst. 9, 671-7.

51. Harding, T. T., "Low-Temperature Apparatus for Single-Crystal X-Ray Goniometers, Can. J. Chem. 34, 371-5.

52. Kuznetsov, V. G., "The X-Ray Analysis at High and Low Temperatures," Zhur. Neorg. Khim. 1, 1548-58.

53. Pavlovic, A. S., "Design and Application of a Weissenberg X-Ray Camera for Studies Near Liquid Helium Temperature," Univ. Microfilms (Ann Arbor, Mich.) Publ. No. 16725, Dissertation Abstr. 16, 1156.

1957

54. Black, I. A. and Brooks, F. P., "A Liquid Helium Cold Cell for Use with an X-Ray Diffractometer," 15th Annual Pittsburgh Diffraction Conference.

55. Eeles, E. G., "Precision Low-Temperature X-Ray Camera," Proc. Cryogenic Eng. Conf., 2nd, Boulder, 248-53.

56. Jetter, L. K., McHargue, C. J., Williams, R. O. and Yakel, H. L., Jr., "Low Temperature Camera for X-Ray Diffractometer," Rev. Sci. Instr. 28, No. 12, 1087-8.

57. Simmons, R. O. and Balluffi, R. W., "Low Temperature Thermal Expansion of Copper," Phys. Rev. 108, No. 2, 278-80.

1958

58. Black, I. A., Bolz, L. H., Brooks, F. P., Mauer, F. A. and Peiser, H. S., "A Liquid-Helium Cold Cell for Use with an X-Ray Diffractometer," J. Research Nat. Bur. Stand. 61, No. 5, 367-71.

59. Fridrichsons, J. and Mathieson, A. McL., "Low Temperature Attachment for a Single Crystal Equi-inclination Weissenberg Goniometer," Rev. Sci. Instr. 29, No. 9, 784-5.

60. Guengant, L., "Cryostat for X-Ray Diffraction Studies of Ice at High Pressures," J. Rech., No. 45, 371-3.

61. Hutchinson, T. E. and Miller, J. E., "A Low Temperature X-Ray Diffraction Camera," Bull. Amer. Phys. Soc. 3, No. 4, 304.

62. Zevin, L. S. and Kheiker, D. M., "Attachment to the URS-SOI Apparatus for Exposures at High and Low Temperatures," Zavodskaya Lab 24, 636-8.

1959

63. Aliev, N. A. and Ibragimov, N. I., "Vacuum Camera for Low and High Temperature," Doklady Akad. Nauk Azerbaidzhan S. S. R. 15, No. 4, 289-92.

64. Baun, W. L., "Simple Low Temperature Diffractometer Specimen Mount," Appl. Spectros. 13, 80.

65. Bolz, L. H., Mauer, F. A. and Peiser, H. S., "Exploratory Study by Low-Temperature X-Ray Diffraction Techniques of Diborane and the Products of a Micro-wave Discharge in Diborane," J. Chem. Phys. 31, 1005-7.

66. Gräicher, H., Helg, U. and Schär, R., "Röntgen Camera for Very Low Temperatures," Helv. Phys. Acta, 32, 474-6.

67. Neynaber, R. H., Brammer, W. G. and Beeman, W. W., "Mechanism of the Small-Angle X-Ray Scattering from Cold-Worked Metals," J. Appl. Phys. 30, 656-61.

68. Viswamitra, M. A., "X-Ray Camera for Study of Crystals at Low Temperature," Proc. Indian Acad. Sci., Sect. A, 49, No. 6, 326-32.

1960

69. Taylor, A., "A Versatile 19-cm Diameter Low-Temperature Debye-Scherrer Camera," Adv. in X-Ray Anal. III. Proc. 8th Annu. Conf. Appl. X-Ray Anal., Estes Park (1959). New York, Plenum Press. 23, 5X16, 41-7.

70. Attard, A. E. and Azaroff, L. V., "Liquid-Nitrogen Cryostat for Single Crystal X-Ray Diffraction," J. Sci. Instr. 37, 238-39.

71. Viswanitra, W. A. and Ramaseshan, S., "Simple Device for Growing Crystals at Low Temperatures in X-Ray Camera," Rev. Sci. Instr. 31, No. 4, 456-7.

72. Wagner, C. N. J., "Stacking Faults in Gold after a Cold-Work Distortion at Low Temperature," Z. Metallk. 51, 259-64.

1961

73. Barrett, C. S., "X-Ray Diffraction Studies at Low Temperatures," 10th Annu. Conf., Appl. X-Ray Anal., Denver Colo.

74. Beckman, O., "Magnetic Properties of $KMnF_3$. I. Crystallographic Studies," Phys. Rev. 121, 376-80.

75. Weltman, H. J., "Low Temperature X-Ray Diffraction of Frozen Electrolytes," 10th Annu. Conf. Appl. X-Ray Anal., Denver, Colo.

1962

76. Klug, H. P. and Sears, D. R., "Solid State Studies of the Noble (Rare) Gases and Their Solid Solutions," AFOSR Contract AF(638)-575, Solid State Sciences Div., AF Office of Scientific Research, Wash 25, D.C.

Reviews

77. Ruhemann, M. and Ruhemann, B., "Low Temperature Physics," Cambridge University Press, 1937.

78. Peiser, H. S., Rooksby, H. P. and Wilson, A. J. C., "X-Ray Diffraction by Polycrystalline Materials," Chapter 10 Low Temperature Methods by E. G. Steward. The Institute of Physics, London, 1955.

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